
टार और बिटुमन सामग्रियों के लिए परीक्षण
पद्धतियाँ — विस्कासिता ज्ञात करना

भाग 2 निरपेक्ष विस्कासिता

(दूसरा पुनरीक्षण)

Methods for Testing Tar and
Bituminous Materials —
Determination of Viscosity

Part 2 Absolute Viscosity

(Second Revision)

ICS 75.140

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FOREWORD

This Indian Standard (Second Revision) was adopted by the Bureau of Indian Standards after the draft finalized by the Bitumen, Tar and Related Products Sectional Committee, had been approved by the Petroleum, Coal and Related Product Division Council.

This standard was originally published in 1958 as 'Methods for testing tar and bituminous materials — Determination of viscosity: Part 2 Absolute viscosity' and subsequently revised in 1978. 'Methods for testing tar and bituminous materials' was originally published as series of 22 standards in the form of a booklet, as listed below:

<i>IS No.</i>	<i>Title</i>
1201 : 2004	Sampling
1202 : 1978	Determination of specific gravity
1203 : 1978	Determination of penetration
1204 : 1978	Determination of residue of specified penetration
1205 : 1978	Determination of softening point
1206 (Part 1) : 1978	Determination of viscosity: Part 1 Industrial viscosity
1206 (Part 2) : 1978	Determination of viscosity: Part 2 Absolute viscosity
1206 (Part 3) : 1978	Determination of viscosity: Part 3 Kinematic viscosity
1207 : 1978	Determination of equiviscous temperature (EVT)
1208 : 1978	Determination of ductility
1209 : 1978	Determination of flash point and fire point
1210 : 1978	Float test
1211 : 1978	Determination of water content dean and stark method
1212 : 1978	Determination of loss on heating
1213 : 1978	Distillation test
1214 : 1978	Determination of matter insoluble in benzene (WITHDRAWN due to toxic nature of benzene)
1215 : 1978	Determination of matter insoluble in toluene
1216 : 1978	Determination of solubility in carbon disulphide trichloroethylene
1217 : 1978	Determination of mineral matter ash
1218 : 1978	Determination of phenols
1219 : 1978	Determination of naphthalene
1220 : 1978	Determination of volatile matter content

However, the Committee responsible for the formulation of standards in the field of bitumen, tar and related products decided to publish these Indian Standards separately for each test so as to make it user friendly.

(Continued on third cover)

Indian Standard

METHODS FOR TESTING TAR AND BITUMINOUS MATERIALS — DETERMINATION OF VISCOSITY

PART 2 ABSOLUTE VISCOSITY

(*Second Revision*)

1 SCOPE

This standard (Part 2) covers the method for the determination of absolute viscosity of bitumen and cut-backs by vacuum capillary viscometers at any specified temperature. It is applicable to materials having a viscosity range of 42 to 200 000 Poises.

NOTE — The standard covers three types of viscometers and anyone of them can be used for determination of absolute viscosity.

2 REFERENCES

The following standards contain provisions, which through reference in this text, constitute provisions of this draft standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

<i>IS No.</i>	<i>Title</i>
334 : 2002	Glossary of terms relating to bitumen and tar (<i>third revision</i>)

3 TERMINOLOGY

For the purpose of this standard, the following definitions and those given in IS 334 shall apply.

3.1 Absolute or Dynamic Viscosity of a Newtonian Liquid — The ratio between the applied shear stress and rate of shear is called the coefficient of viscosity. This coefficient is thus a measure of the resistance to flow of the liquid. It is commonly called the viscosity of the liquid. The SI unit of viscosity is $1 \text{ Pa} \cdot \text{s}$ ($1 \text{ N} \cdot \text{s}/\text{m}^2$) and is called a Pascal-second. The cgs unit of viscosity is $1 \text{ g}/\text{cm} \cdot \text{s}$ ($1 \text{ dyne} \cdot \text{s}/\text{cm}^2$) and is called a poise (P). $1 \text{ Pa} \cdot \text{s}$ is equivalent to 10 P.

3.2 Newtonian Liquid — A liquid in which the shear stress is directly proportional to the rate of shear. The constant ratio of shear stress to the rate of shear is called

the coefficient of viscosity of the liquid. If this ratio is not constant then the liquid is non-Newtonian.

4 APPARATUS

4.1 Viscometers — Capillary type made of borosilicate glass, annealed suitable for this test are given in 4.1.1 to 4.1.3.

4.1.1 Cannon-Manning Vacuums Viscometer (Fig. 1) — The size numbers/approximate bulb factors *K*, and viscosity ranges for the series of Cannon-Manning Vacuum Capillary Viscometer are as follows:

Viscometer Size No.	Approximate Calibration Factor 30 cm Hg Vacuum Poises per s		Viscosity Range Poises
	Bulb B	Bulb C	
10	2.0	0.6	36 to 800
11	6.0	2.0	120 to 2 400
12	20.0	6.0	360 to 8 000
13	60.0	20.0	1 200 to 24 000
14	200.0	60.0	3 600 to 80 000

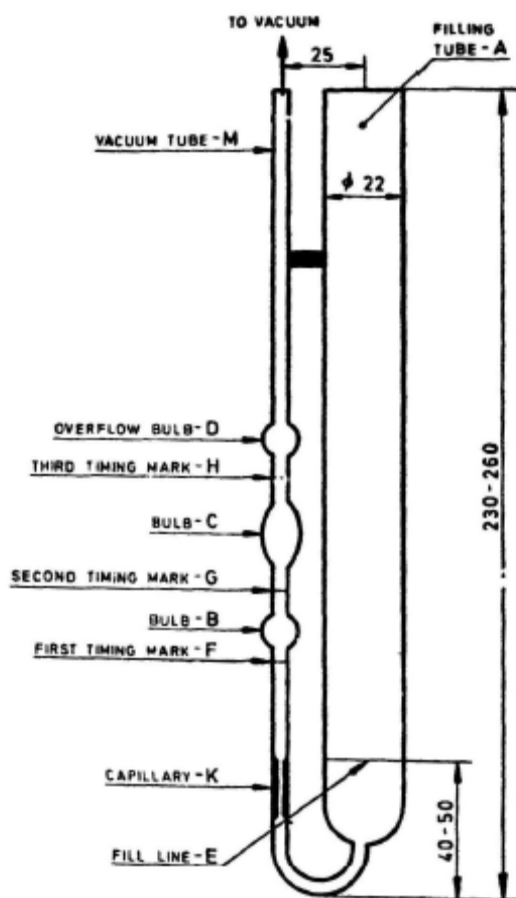
For all viscometer sizes the volume of measuring bulb C is approximately three times that of bulb B. The viscosity ranges correspond to a filling time of 60 and 400 s for both measuring bulbs.

Sizes 10 through 14 are best suited to viscosity measurements of bituminous binders at 60 °C.

NOTE — The calibration factors have to be determined either by calibration through viscosity standards or through calibration by competent agency.

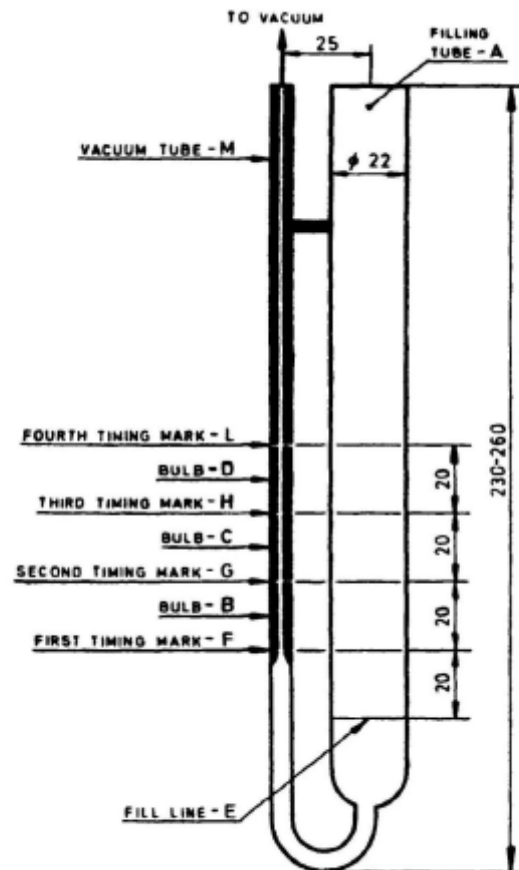
4.1.2 Asphalt Institute Vacuum Viscometer (Fig. 2) — The size numbers, Approximate radii, approximate bulb factors *K*, and viscosity ranges for the series of Asphalt Institute Vacuum Capillary Viscometer are as follows:

Viscometer Size No	Capillary Radius K. cm	Approximate Calibration Factor 30 cm Hg. Vacuum: Poises per s			Viscosity Range Poises
		Bulb B	Bulb C	Bulb D	
25	0.0125	2	1	0.7	42 to 800
50	0.025	8	4	3	180 to 3 200
100	0.050	32	16	10	600 to 12800
200	0.100	128	64	40	2400 to 52000
400	0.200	500	250	160	9600 to 200 000



All dimensions in millimetres

FIG. 1 CANNON-MANNING VACUUM CAPILLARY
VISCOMETER



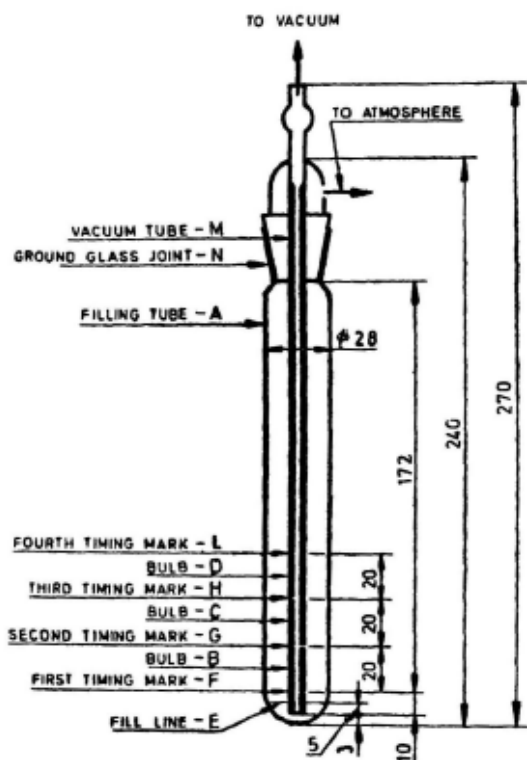
All dimensions in millimetres

FIG. 2 ASPHALT INSTITUTE VACUUM CAPILLARY
VISCOMETER

This viscometer has measuring bulbs *B*, *C* and *D* located on the viscometer arm *M* which is a precision bore glass capillary. The measuring bulbs are 2 cm long capillary segments separated by timing marks *F*, *G*, *H* and *L*.

Sizes 50 through 200 are best suited to viscosity measurements of bituminous binders at 60 °C.

Note: The calibration factors have to be determined either by calibration through viscosity standards or through calibration by competent agency.



All dimensions in millimetres

FIG. 3 MODIFIED KOPPERS VACUUM CAPILLARY VISCOMETER

4.1.3 Modified Koppers Vacuum Viscometer (Fig. 3) — The size numbers approximate radii, approximate bulb factors *K*, and viscosity ranges for the series of modified Koppers vacuum capillary viscometer are as follows:

Viscometer Size No.	Capillary Radius cm	Approximate Calibration Factor <i>K</i> . 30 cm Hg. Vacuum: Poises per s			Viscosity Range Poises
		Bulb B	Bulb C	Bulb D	
25	0.0125	2	1	0.7	42 to 800
50	0.025	8	4	3	180 to 3200
100	0.050	32	16	10	600 to 12800
200	0.100	128	64	40	2 400 to 52 000
400	0.200	500	250	160	9 600 to 200 000

Sizes 50 through 200 are best suited to viscosity measurements of bituminous binders at 60 °C.

NOTE — The calibration factors have to be determined either by calibration through viscosity standards or through calibration by competent agency.

This viscometer consists of a separate filling tube *A*, and a precision bore glass capillary vacuum tube *M*. These two parts are joined by borosilicate ground glass joint *N*, having a 24/40 standard taper. The measuring bulbs *B*, *C*, and *D* on the glass capillary are 2 cm capillary segments separated by timing marks *F*, *G*, *H* and *L*.

A viscometer holder can be made by drilling a 28 mm hole through the center of a No. 11 rubber stopper and setting the stopper between the hole and the edge. When placed in a 5 cm diameter hole in the bath cover, it holds the viscometer in place.

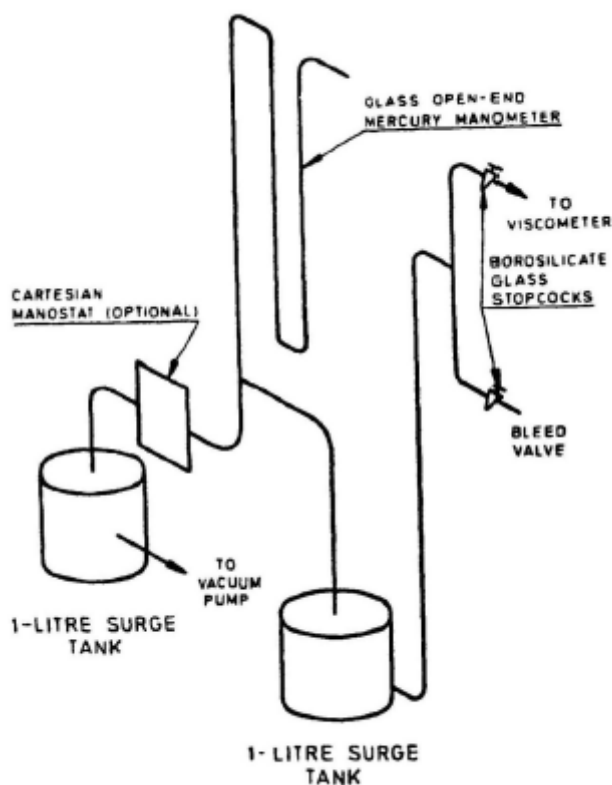
4.2 Thermometer — The thermometer shall be calibrated from a competent agency with least count of 0.1 °C

4.3 Bath — A suitable bath for immersion of the viscometer so that the liquid reservoir or top of the capillary, whichever is uppermost is at least 20 mm below the upper bath level, and with a provision for the visibility of the viscometer and the thermometer. Firm support for the viscometer shall be provided. The efficiency of the stirring and the balance between heat losses and heat input must be such that the temperature of the bath medium does not vary by more than ± 0.1 °C.

4.4 Vacuum System — A vacuum system capable of maintaining a vacuum to within ± 0.05 cm of the desired level up to and including 30 cm of mercury. One such system is shown in Fig. 4. The glass tubing of 6.35 mm diameter and all glass joints should be completely airtight and no loss of vacuum should be permitted till the experiment is on. A vacuum or aspirator pump is suitable for the vacuum source.

NOTE — The vacuum measuring system for this test method must be standardized at least once a year.

4.5 Timing Device — A Stop watch or other timing device graduated in divisions of 0.2 s or less, and accurate to within 0.1 percent when tested over a 60 min period.



NOTE — All tubing is of glass with 6.35 mm OD

FIG. 4 SUGGESTED VACUUM SYSTEM FOR VACUUM CAPILLARY VISCOMETERS

5 CALIBRATION OF VISCOMETER

5.1 Reference Material — Viscosity standard (certified viscosity reference standard) may be used for calibration purposes.

5.2 Calibration — Charge a clean-dry viscometer by pouring the reference material to within ± 2 mm of fill line *E* (Fig. 1, 2 and 3). Place the charged viscometer in the viscometer bath maintained within ± 0.1 °C at the calibration temperature. Establish a 30 ± 0.05 cm vacuum in the vacuum system and connect it to the viscometer with valve closed in the line leading to the viscometer. After the viscometer has been in the bath for 30 ± 5 min, start the flow of liquid in the viscometer by opening the stop cock in the line leading to the vacuum system. Measure to within 0.5 s the time required for the leading edge of the meniscus to pass between timing marks *F* and *G*. Also measure to within 0.5 s the time required for the leading edge of the meniscus to pass between timing marks *G* and *H*. Calculate the calibration

factor *K* for each bulb as follows:

$$K = V/t$$

where

K = viscometer bulb calibration factor poises/s at 30.0 cm Hg;

V = absolute viscosity of reference material at calibration temperature in poises; and

t = flow time, in seconds.

Repeat the calibration procedure using the same viscosity standard or another reference material. Record the average calibration constant *K*.

5.2.1 The duplicate determination of calibration constant *K* for each bulb shall be within 2 percent of the mean value. The value of viscometer constants shall be expressed to the nearest 0.1 percent.

6 PROCEDURE

6.1 Preparation of the Sample

Heat the sample to a temperature not more than 60 °C for the tars and pitches and not more than 90 °C for bitumen above their respective approximate softening point temperature respectively until it has become sufficiently fluid to pour. Transfer about 20 ml into

a suitable container and maintain it to a temperature of 135 ± 5.5 °C stirring occasionally to prevent local overheating and allow the entrapped air to escape.

6.1.1 Charge the viscometer by pouring the prepared sample to within ± 2 mm of fill line *E*. Place the charged viscometer in an oven or bath maintained at 135 ± 5.5 °C for a period of 10 ± 2 min to allow large air bubbles to escape.

6.2 Testing

Maintain the bath at the test temperature within ± 0.1 °C. Place the charged viscometer vertically in the water bath with the help of a holder so that the uppermost timing mark is at least 2 cm below the surface of the bath liquid. Establish a vacuum of 30 ± 0.05 cm of mercury in the vacuum system and connect it to the viscometer with the valve closed. After the viscometer has remained in the bath for 30 ± 5 min open the valve and allow the asphalt to flow into the viscometer. Measure to within ± 0.5 s the time required for the leading edge of the meniscus to pass between successive pairs of timing marks. Upon completion of the test, remove the viscometer from the bath and place it in an inverted position in an oven maintained at 135 ± 5 °C until asphalt is drained off thoroughly from the viscometer. Clean the viscometer thoroughly by rinsing several times with an appropriate solvent completely. Dry the tube by passing a slow stream of filtered dry air through the capillary for 2 min. Periodically clean the instrument with chromic acid to remove organic deposits. Rinse thoroughly with distilled water and acetone and dry with clean air.

7 CALCULATION

7.1 Calculate and report the absolute viscosity to by the following equation:

$$\text{Viscosity (in Poises)} = Kt$$

where

K = selected calibration factor, in poise per second; and

t = flow time, in seconds.

NOTE — Measure the time required for the leading edge of the meniscus to pass between successive pairs of timing marks. Report the first flow time which exceeds 60s between a pair of timing marks, noting the identification of the pair of timing marks.

7.2 Always report the test temperature and vacuum with the viscosity test results. For example, viscosity at 60 °C, 30 cm Hg vacuum in poises.

8 PRECISION

8.1 The duplicate test results should not differ by more than the following:

- a) *Repeatability*: The duplicate test results by the same operator using the same viscometer should not differ by more than 7 percent of their mean.
- b) *Reproducibility*: Results obtained by two laboratories should not differ by more than 10 percent of their mean.

Please also note that this precision is only for meant for measurement made at 60 °C and hence should not be used for any other temperature measurements.

ANNEX A

(Foreword)

COMMITTEE COMPOSITION

Bitumen, Tar and Related Products Sectional Committee, PCD 06

<i>Organization</i>	<i>Representative(s)</i>
CSIR-Central Road Research Institute, New Delhi	PROF SATISH CHANDRA (Chairman)
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Hindustan Petroleum Corporation Limited, Mumbai	SHRI SANTOSH DHAKU BHOGALE
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Indian Institute of Technology Bombay, Mumbai	DR DHARAMVEER SINGH
Indian Institute of Technology Delhi, New Delhi	DR ARAVIND SWAMY

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Member Secretary

SHRIMATI D. UMA
SCIENTIST 'D' (PCD), BIS

(Continued from second cover)

Accordingly, second revision of the standard was taken up to formulate individual standard on determination of Absolute Viscosity. The use of suitable thermometers has been permitted in the place of mercury in glass type thermometer mentioned in the earlier version of the standard. The terms viscosity and coefficient of viscosity have been redefined. Calibration of Viscometers with viscosity standards has been recommended. Recommendations regarding the sizes of viscometers suitable for viscosity measurements of bituminous binders at 60 °C have been made.

The Composition of the Committee responsible for formulation of this standard is given at Annex A.

In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*).

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Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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